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DOLOMITE-BASE REFRACTORIES

BY

ALVIN SCHALLIS

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UNITED STATES DEPARTMENT OF THE INTERIOR - BUREAU OF MINES

DOLOMITE-BASE REFRACTORIES[1/]

By Alvin Schallis[2/]

INTRODUCTION

Of all the uses for dolomite that depend on its magnesia content, perhaps the most important is its use in basic refractories. The quantities of dolomite (including both that used as crude and that calcined before use) sold or used for refractory purposes by producers in the United States for 1920-41, inclusive, are shown in figure 1. As can be seen from the chart, the utilization of dolomite as a basic refractory is far from new; it was, in fact, the first basic refractory used extensively in open-hearth steel furnaces. After large deposits of magnesite were opened in Austria-Hungary, the use of dolomite in the open hearth declined somewhat, only to be actively revived during the World War of 1914 to 1918, when supplies from Austria were shut off. The use of dolomite as a refractory in the steel industry again declined slightly for a brief period after the Armistice, but subsequently its use has tended steadily to increase, latest statistics indicating that more dead-burned dolomite is used per ton of open-hearth steel now than during the World War of 1914 to 1918. Despite fluctuations in consumption, dolomite has continued to be by far the most widely used basic refractory material. The following table, showing the quantities of basic refractories consumed in the manufacture of steel, the principal outlet, indicates the relative importance of dolomite.

The chief reason for the widespread use of dolomite as a basic refractory is its relatively low cost. Its abundance and the proximity of the deposits to the steel industry (see fig. 2) afford every incentive to enhance the desirable physical properties of dolomite refractories and to ameliorate certain of their less desirable characteristics. The present emergency, with its huge demands upon the steel industry, has lent further impetus to the work of improving dolomite refractories.

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Average quantities of basic refractories used in the production of basic open-hearth steel for a pre-war and post-war period 1/

	Weighted average quantity used per gross ton of basic open-hearth steel, pounds	
	1910-1914	1923-1932
Dead-burned grain magnesite	7.68	2.55
Magnesite brick	1.48	.97
Chrome ore and chrome brick	2.39	3.26
Raw dolomite	22.01	33.88
Calcined and dead-burned dolomite	31.07	45.67
Total refractories (excluding silica and clay)	64.63	86.33

1/ Data from the records of producers of 72 percent of the basic open-hearth steel output of the country. Consumption figures for each company have been weighted by its production of basic open-hearth steel (1923-32).

One immediate objective has been the production of a basic refractory, manufactured largely from dolomite, that would be comparable with or superior to present magnesite refractories and that could be supplied at equal or lower cost. Such a development would assure essential metallurgical industries in the eastern United States of adequate supplies of basic refractories for all time regardless of possible closing of sea lanes or difficulties of transcontinental railroad deliveries from domestic magnesite deposits in California, Washington, and Nevada. The freight rate alone on domestic magnesite to the Pittsburgh, Pa., consuming area is $14.40 per short ton for minimum carloads of 80,000 pounds. This is equivalent to 58 to 65 percent of the sales price f.o.b. shipping points in Washington and California. It also costs more to mine magnesite than dolomite, because dolomite deposits are more nearly uniform, and virtually no hand-sorting or mechanical concentration is needed. In view of the marked advantage in such fundamental cost factors, it is surprising that more intensive efforts to make dolomite refractories more nearly comparable to magnesite refractories were not undertaken long ago.

Before considering the refractory properties of dolomite, it may be well to list some of the requirements that must be met by all refractories. First, and foremost, is the requirement that the softening point or the temperature at which the refractory loses its mechanical strength must be high enough; this is obvious, as the softening point limits the temperature at which the refractory may be used. An apparent exception to this is the use of grain refractories, which are employed mainly for furnace bottoms and which (because of the lower strength requirements and because a certain degree of plasticity is actually desirable) are used at relatively higher temperatures with respect to the softening point than are brick and other preformed refractories,

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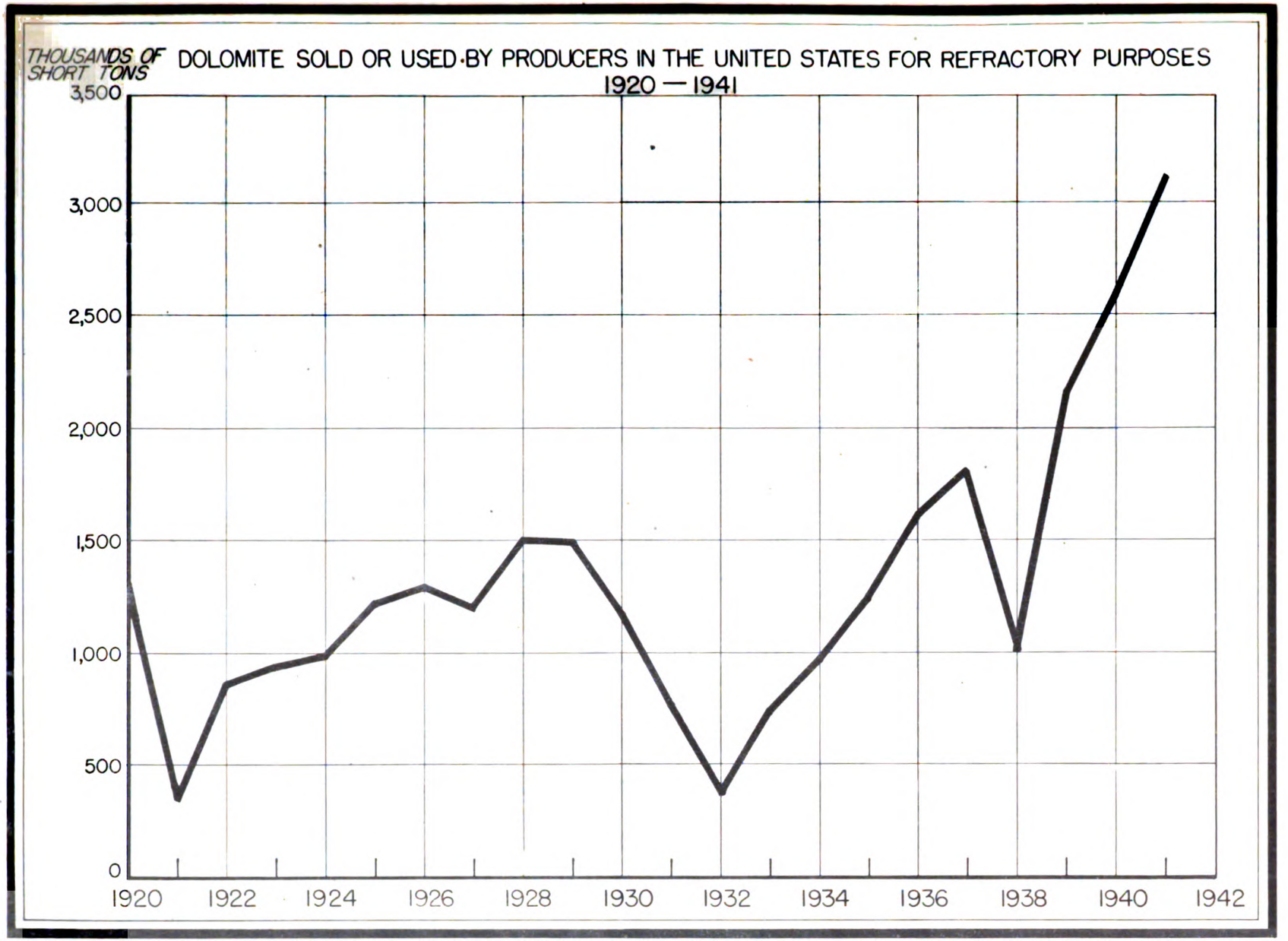

Figure 1.

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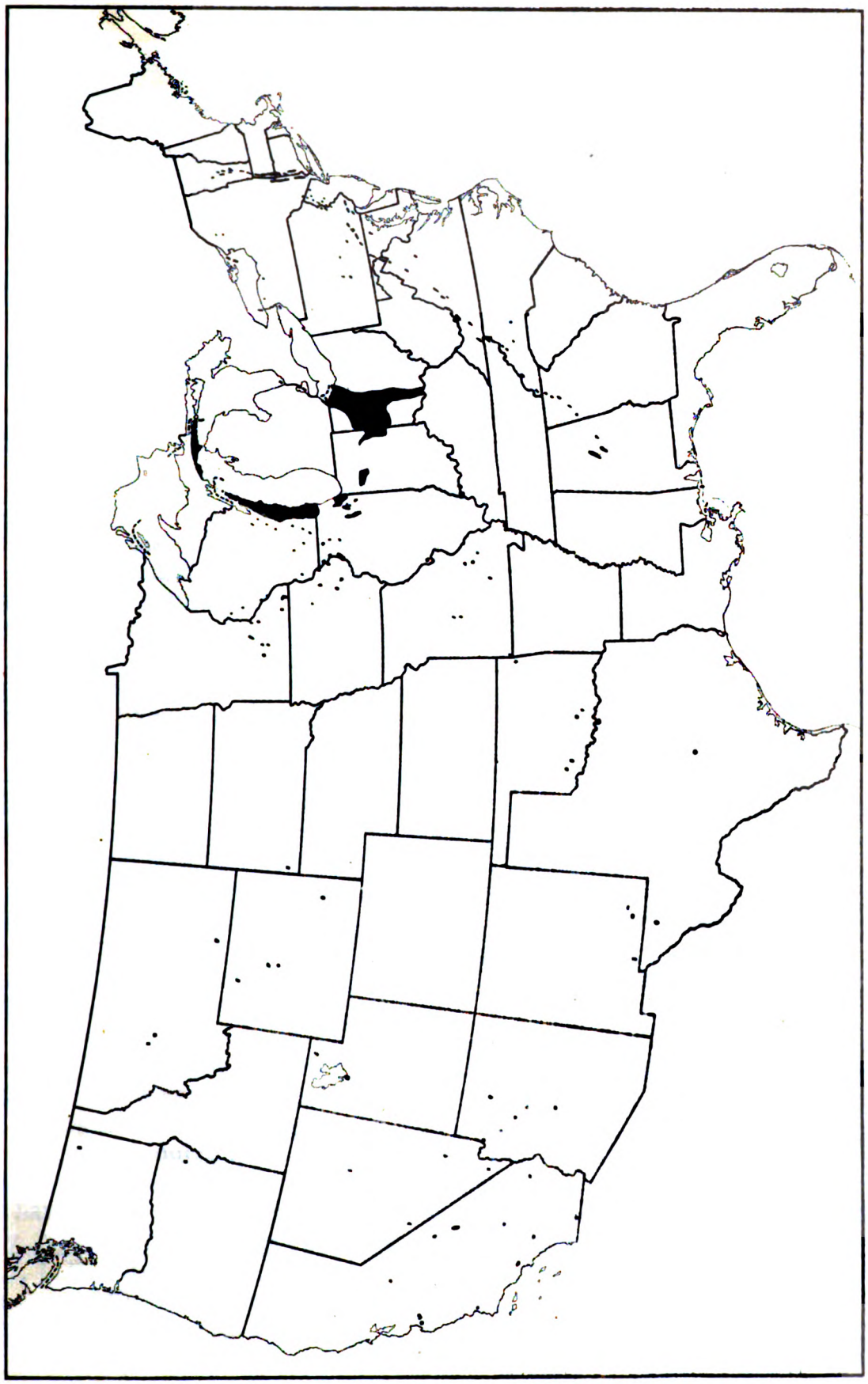

Figure 2.- High-purity dolomite deposits in the United States.

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which are likely to be subjected to tension or shear. Second only to resistance to high temperatures is the requirement that a refractory must resist attack by the materials with which it comes in contact. It is often impractical or impossible to obtain a refractory material that is completely resistant to its environment; consequently, materials that are attacked slowly are often used. This restriction applies not only to high-temperature environment but also to low-temperature atmospheric environment in which moisture and carbon dioxide are factors. The above qualities - resistance to high temperature and resistance to contact materials - are the essential requirements of a refractory. Other highly desirable characteristics are resistance to spalling, low change in volume with change in temperature, low porosity, low permeability to furnace products (gaseous, liquid, or dusty), suitable electrical conductivity (usually low), suitable heat-conducting characteristics (generally low), and low cost.

CALCINED DOLOMITE REFRACTORIES

Calcining is the chief operation in the usual preparation of dolomite as a refractory, for although raw dolomite is often spread on the banks of the open-hearth steel furnace, this material is quickly calcined by the heat of the furnace. According to Conley[3/], dolomite decomposes with the evolution of CO_2 at about 725°C. (1,337°F.). Upon being heated to this temperature, the mineral breaks down into calcite and magnesite, and the magnesite (which, had it been previously free, would have decomposed at a lower temperature) immediately releases its CO_2 causing the formation of extremely fine periclase. Upon continued heating, the calcite breaks down into lime and CO_2 at about 900°C. (1,650°F.), and lime exists in the presence of the periclase without further reaction. The system CaO-MgO is a simple binary eutectic system with a eutectic melting point of 2,300°C. (4,170°F.) and a eutectic composition (as reported in the international Critical Tables) of 67 percent CaO and 33 percent MgO (the theoretical composition of calcined pure dolomite is 58.4 percent CaO and 41.6 percent MgO). It will be noted that even the eutectic melting point of this system is considerably above any temperatures likely to be encountered in ordinary metallurgical practice.

Chief among the objections to the use of pure dolomite as a refractory is the high degree of reactivity of its basic oxides. However, merely by heating the dolomite for a sufficient length of time to temperatures substantially higher than those usually required for simply driving off the CO_2, the periclase is recrystallized into relatively coarse granules that present little surface and therefore have a low rate of reaction in the usual environments encountered. Ordinarily, however, it is impractical to heat the dolomite to the temperatures

3/ Conley, J. E. Calcination Conditions for Limestone, Dolomite, and Magnesite: Am. Inst. Min. and Met. Eng. Tech. Paper 1037, H-75; Min. Technol., March 1939, 15 pp.

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required to recrystallize the lime and render it essentially inert. Pure calcined dolomite, therefore, cannot be stored where it is exposed to a moist atmosphere for more than a few days without appreciable deterioration. Improper storage causes the lime to become air-slacked, and the material soon falls to a powder. Even the periclase may be somewhat unstable in storage, but when properly burned it will resist deterioration for months or even years. Attempts have been made to coat the grains of burned dolomite with tar to protect them from the atmosphere until they are rammed into or applied to the furnace, but such protection is temporary and is destroyed by the first heat of the furnace. This procedure, therefore, is unusual in American practice.

The high melting point of pure, burned dolomite impedes proper sintering of the grains, so that they will remain in position. In building bottoms of basic open-hearth furnaces, however, this difficulty is overcome by mixing it with substantial quantities (commonly as high as 20 percent) of basic open-hearth slag.

The slag, which consists largely of calcium and iron silicates, fuses and makes the particles of dolomite stick together, later reacting with some of the lime in the dolomite to form the di- and tri-calcium silicates (which are quite refractory) and relatively small quantities of fusible calcium ferrites. These hold the dolomite in place and form a firm bottom.

To aid calcination, to help stabilize the calcium oxide, and to improve its ability to sinter in the open-hearth furnace, iron oxide is generally added to the dead-burned dolomite before the charge goes to the kiln. The amount added, which varies according to the preference of the manufacturer, ranges from 2 to 12 percent, 7 percent being a fair average. Magnesium oxide and iron oxide form a continuous series of solid solutions from periclase (MgO) to magnesium ferrite ($MgO.Fe_2O_3$), the iron oxide first reacting with the periclase at 1,300° to 1,400°C. (2,370° to 2,550°F.). As the temperature is raised above 1,500°C. (2,730°F.), dicalcium ferrite ($2CaO.Fe_2O_3$) is formed from the remaining free iron oxide and the free lime.4/ At the temperatures encountered, however, the equilibrium of the reaction $CaO + MgO.Fe_2O_3 \rightleftharpoons MagO + CaO.Fe_2O_3$ favors the formation of magnesium ferrite. This is largely absorbed by the magnesia to form a solid solution and to facilitate grain growth and dead-burning of magnesia. Some evidence indicates that calcium ferrites may be able to go into solid solution slightly in periclase and behave somewhat similarly to magnesium ferrite.5/ The reactions between ferric oxide and dolomite originate at the surfaces of the particles, but reactants and reaction products gradually diffuse into the dolomite particles until iron compounds are found throughout. However,

4/ Konopicky, K., Sintered Magnesite. II. Reactions During Sintering: Ber. Deut. keram. Ges., vol. 18, No. 3, 1937, pp. 97-106.

5/ Schwiete, H. E., and Strassen, H. Zur, (Properties of Cements Rich in Magnesia): Zement, vol. 25, No. 49, 1936, pp. 843-846, 861-865, 879-882.

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because this diffusion is a relatively slow process, the bulk of the iron compounds remains at or near the surfaces of the dolomite particles and thereby coats them. Inasmuch as the ferrites of calcium and magnesium are more stable to the action of carbon dioxide and moisture than are lime or magnesia, the coating of iron compounds somewhat protects the refractory dolomite from the action of the atmosphere. Dolomite burned in the presence of quantities of iron oxide is consequently more stable than pure burned dolomite.

Another function served by the calcium ferrite coating is to form a well-bonded dolomite surface. The calcium ferrite film on the dolomite begins to soften at about 1,436°C. (2,617°F.), causing the particles to stick together. If this high temperature is maintained, the calcium ferrite gradually reacts with the magnesium oxide and diffuses into the periclase in the interior of the particle, thereby stiffening the surface film and strengthening the bond. Similarly, the silicates of admixed basic open-hearth slag also tend to react with the lime of the low-melting calcium ferrite, forming refractory dicalcium silicate and permitting the iron oxide to react further with the magnesium oxide. Thus, a satisfactorily refractory hearth surface is formed.

The presence of the iron oxide in solid solution tends to disturb the forces in the crystal lattice and thereby facilitates recrystallization and grain growth of the periclase (and to some extent the lime, also), which in turn causes shrinkage and increased density because of the filling of the voids left by the expulsion of CO_2. The shrinkage of dolomite due to the heat treatment, amounting to approximately 20 percent of the original particle diameters, together with attrition in the rotary kiln, results in a reduction in particle size on passage through the kiln of about one screen interval on the Tyler standard series. The particles retain approximately their original shapes during the process.

The particle-size distribution of refractory materials is very important. It is obvious that the less porous the structure, the greater the strength. To reduce the volume of the voids between grains and to increase the density of the refractory, an effort usually is made to have such a distribution of particle sizes that the small particles will just fill the interstices between the larger grains. Owing to the blocking effect of intermediate particles on the flow of the finer material between the larger particles, it is usually found that maximum density cannot be obtained with grain materials having a continuous distribution of particle sizes from the coarsest to the finest. Therefore, some manufacturers of refractories use the "three-screen" process to obtain a "gap-size" product from which the very coarse and intermediate particles have been removed and in which a proper blend of coarse and fine particles has been made. Obviously, such a process involves considerable expense that would not be justified in the preparation of a low-cost refractory such as dolomite. When

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dolomite is used, therefore, an effort is made to facilitate the natural compaction of the material to maximum density by keeping the particle sizes within definite limits, and this is accomplished by selecting and adjusting the crushing and grinding equipment to yield the most favorable distribution of particle sizes. Extremely fine particles are removed to avoid the formation of objectionable dust during handling of the dolomite, but their removal may also improve the free-flowing properties of the grain material. Dolomite refractories having particle sizes from through 3/8- by 3/4-inch rectangular mesh and on 3/16-inch mesh to material through 3/16-inch mesh and on 12-mesh are quite usual.

The refractory dolomite just described, though reasonably low-priced, leaves much to be desired in the way of refractory and handling properties. As mentioned previously, it is somewhat unstable to atmospheric conditions, but even more objectionable than the moderate deterioration of refractory properties is the slight degree of surface slaking that occurs during relatively short periods of storing. This produces quantities of dry, dusty calcium hydroxide, which becomes very objectionable in handling the material. Several manufacturers add about 1 gallon of fuel oil per ton to the freshly manufactured calcined dolomite primarily to lay the dust. The oil treatment also improves the stability of the product slightly in that it tends to close the pores and prevents access of moisture and carbon dioxide to the interior of the particles.

A few basic open-hearth bottoms constructed from calcined dolomite have been operated successfully over considerable periods with virtually no interruption. Dolomite is less costly than magnesite, but the threat of trouble through reactivity of the lime is great enough to discourage widespread use of calcined dolomite for furnace bottoms. Refractory dolomite is therefore used generally for the maintenance of the open-hearth working surface and for minor repairs rather than for the construction of the hearth. During the life of a basic open-hearth furnace, however, the quantity of refractory dolomite used in maintaining the working surface on the hearth and in minor repair work is many times as great as would be used in the construction of the actual hearth.

Calcined dolomite, with or without iron oxide, is generally too unstable for the manufacture of satisfactory refractory brick. In Europe, some protected dolomite refractory bricks have been manufactured and used, but they have never been popular. Numerous methods have been employed to protect dolomite brick and other special shapes from atmospheric attack during storage. Some of them have been partly successful in permitting storage for a few weeks or even a few months. These methods involve impregnating the brick with a water-repelling material, such as paraffin or tar, or encasing the brick with a water-proof covering, such as a treated paper. These protective coverings, although aiding the storing qualities before use, are destroyed the first time the brick is sub-

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jected to high temperatures, and thereafter the brick is no longer protected. Any shut-down of the furnace will therefore permit deterioration of the brick.

STABILIZED DOLOMITE REFRACTORIES

Inasmuch as the chief objection to dolomite refractories is the lime content, the logical remedy is either to remove the lime or to change it by chemical reaction into an unobjectionable calcium compound. Removal of the lime leaves essentially a magnesia refractory comparable with refractories prepared from magnesite. Rendering the lime inactive, the method that has first achieved commercial success, not only conserves the superior qualities of the magnesia but also offers the possibility of improving the product by adding a calcium compound having desirable qualities. If the lime of the dolomite is not removed, the calcium compound constitutes so large a part of the refractory that it also must have adequate refractory properties and, in addition, must be compatible with the periclase present. Furthermore, the material added to react with the lime should be cheap and readily available, and the process by which the alteration is effected should be simple and easily accomplished.

It has been found that the conversion of the lime into di- and tri-calcium silicate by the addition of siliceous material is the most satisfactory solution of this problem. Unfortunately, however, tri-calcium silicate is unstable, breaking down into lime and beta-dicalcium silicate, which itself is unstable below 675°C. (1,247°F.) and reverts at low temperatures to the gamma form, with a 10-percent increase in volume. This expansion causes it to fall to a powder and thus renders it unsuitable for refractory purposes. It is possible, however, to stabilize the beta form or at least to inhibit its inversion to the gamma form by the addition of certain materials, notably chromite, phosphates, or borates, which serve as negative catalysts. Chromite is perhaps the most useful of these stabilizers. Borates are objectionable, inasmuch as they reduce materially the refractory properties of the system; and although little is known about the action of phosphates, it is known that phosphates soon lose their efficiency.

To be effective, the stabilizer must be present before the dicalcium silicate is formed. When chromite is used, a small quantity of calcium chromate is formed by reaction with free lime, and this probably goes into solid solution in the beta-dicalcium silicate during its formation.

Iron and aluminum oxides also tend to stabilize dicalcium silicate in the beta form but are less effective than are the other three substances mentioned. Because iron oxide is very cheap, however, it is employed as the stabilizer in the usual dicalcium silicate-periclase product rather than chrome ore, which is employed only in special cases.

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Holmes, McCaughey, and Bole[6/] studied the system $CaMg(CO_3)_2$-Fe_2O_3-SiO_2 with reference to the stable refractory field. They concluded that at concentrations of silica greater than 5 percent dolomite is free from slaking, whereas at silica concentrations lower than 8 to 11 percent (depending on the Fe_2O_3 content, the more Fe_2O_3 the less silica may be present) the dolomite will not dust. To produce a stable material, however, small but definite quantities of iron oxide or alumina must be present. Under commercial manufacturing conditions it was found that these equilibrium conditions were not reached readily and that the addition of small quantities of soda ash or other flux was necessary to promote the reaction. The increased temperatures of modern kiln operation and the larger quantities of iron oxide used as flux, however, have greatly facilitated the desired reactions that are now made use of in the manufacture of refractories.

Additions of silica and stabilizer to the dolomite reduce the periclase content by dilution. The calcium silicates in an open hearth are often acted upon by the constituents of the charge, thereby becoming somewhat soft under operating conditions. Sufficient periclase must therefore be present in the refractory to prevent the mass from becoming too soft and thus too readily subject to destruction in the furnace. A periclase content of 60 percent or more affords sufficient resistance to deformation to produce good refractory properties for furnace hearths. It seems reasonable to suppose that the minimum satisfactory periclase content may be varied somewhat according to the nature of the matrix, a matrix that tends to have great affinity for the periclase and to cause its complete dispersion being less satisfactory than one that permits the intergrowth of the periclase grains. The relationship between the periclase and its matrix probably depends on the impurities contained in the matrix.

If all the lime is converted into dicalcium silicate merely by adding SiO_2, the calcined dolomite will contain less than 32 percent periclase. To obtain more periclase in the product, the necessary SiO_2 is preferably supplied in the form of a magnesium silicate, such as serpentine or olivine. These materials then serve as a cheap source of supplementary MgO. Lime, being a stronger base than magnesia, replaces the magnesia in the silicates. The reaction between a magnesium silicate and lime, however, is not simple, and equilibrium is approached slowly. Moreover, the desired periclase content cannot be obtained by adding magnesium silicates alone; hence more magnesium oxide usually must be added in the form of magnesite or brucite. The composition of a typical stabilized dolomite refractory is given in the following table:

6/ Holmes, M. E., McCaughey, W. J., and Bole, G. A., Dolomite Clinker as a Stable Basic Refractory: Rock Products, July 20, 1929, pp. 59-67

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Composition of typical stabilized dolomite refractory

	Percent
Silica	7.0
Alumina.....	1.0
Ferric oxide ..	7.5
Lime	18.0
Magnesia.....	65.5

Inasmuch as equilibrium conditions are seldom reached, a mineralogical analysis of this refractory would be meaningless. However, enough acidic oxides (SiO_2 and Fe_2O_3) must be present to react with all the lime.

In Canada, where much of the development work on this type of refractory was carried out, a large deposit of magnesitic dolomite is found near Kilmar, Quebec. This material has a satisfactory magnesia: lime ratio and by the simple addition of the desired amount of iron oxide and the proper blending of the component minerals a very satisfactory refractory is produced. A typical analysis of the rock being mined from this source is given below:

Composition of Kilmar magnesitic dolomite[1/]

Chemical composition, percent		Mineralogical composition, percent	
SiO_2	3.5	Serpentine ($3MgO.2SiO_2.2H_2O$)	8
Al_2O_3	.3	Magnesite ($MgCO_3$)	57
Fe_2O_3	.2	Dolomite ($CaCO_3.MgCO_3$)	35
CaO	11.0		
MgO	38.0		
CO_2	46.0		
H_2O	1.0		

1/ Lathe, F. E., Basic Refractories in Canada, 1914 and 1939: Bull. Canadian Inst. Min. and Met., March 1940, pp. 83-99.

In the manufacture of stabilized dolomitic refractories the raw materials are ground to size, thoroughly mixed, and then calcined. Time and temperatures in the kiln are adjusted so that the reactions are not quite completed; if they were carried too far the product would be too refractory. Grain dolomite refractories in which the stabilizing reactions have been stopped at the right point tend to melt and become soft and

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sticky when first subjected to the heat of the furnace, and thus sinter properly. Under furnace conditions, equilibrium is gradually approached, and the mass becomes hard and solid. Although it is unnecessary, operators usually add basic open-hearth slag to such a refractory before use because of the high temperatures required to sinter in a bottom containing only the stabilized dolomitic refractory. Better surfaces probably would be obtained, however, if no additional fluxes were used.

Rammed-in grain refractory hearths never should become completely rigid, as this makes the hearth likely to crack. Enough elasticity is needed for it to follow the thermal expansions and contractions of other parts of the furnace without fracturing. Cracks formed during a shut-down period are difficult to seal upon reheating the furnace when the hearth is completely rigid. This condition of slight plasticity, which is advantageous in the monolithic bottom, is highly undesirable, however, in material for the manufacture of refractory brick, as it would cause weakness at high temperatures, thereby rendering the brick virtually worthless.

Although many bricks have been made, both in this country and in Canada, from dolomite refractory material improved and stabilized as described above, this type of material is used most extensively as a grain refractory for monolithic furnace bottoms.

SEPARATION OF LIME AND MAGNESIA IN DOLOMITE

As previously mentioned, another method of alleviating difficulties arising from the lime content in dolomitic refractories is to get rid of the lime. Separation of the lime and magnesia of dolomite has long been an attractive field for engineers, chemists, and inventors. Numerous reactions have been proposed, but until recently none has been enonomical enough to be used in the manufacture of refractories, and commercial production has been undertaken by only a few firms. In one commercial process, CO_2-free water leaches lime from magnesia in calcined dolomite. In another, calcined dolomite is reacted with magnesium-containing brines or raw sea water, and the magnesia from both the liquid and the dolomite is recovered. In a third process, a solution of ammonium chloride is reacted with calcined dolomite, dissolving the lime as the chloride and precipitating magnesia as the hydroxide.

Quite obviously, considerable political and strategic importance would be attached to any economically sound method for the recovery of a fairly good grade of magnesia from dolomite, inasmuch as such a method would render available almost unlimited supplies of an essential material. (See fig. 2 for locations of high-grade deposits.) Such a development

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would have special importance at present, when most of the foreign sources that had formerly supplied a third or more of the domestic consumption of calcined magnesite have been cut off, and when unprecedented quantities of this commodity are urgently required. Moreover, the large-scale production of magnesia from dolomite would relieve the already overburdened railway system of the necessity of transporting large quantities of refractory and other magnesium-containing material from the Western States to the eastern industrial centers.

CONCLUSION

It is believed that developments in the manufacture of basic refractories from dolomite, which have been described briefly in this circular, foreshadow an era in which it will no longer be necessary to transport refractory magnesite over great distances to important consuming areas, because magnesia refractories of local origin and of equal or superior quality will become available. In this trend, dolomite may become the major source of raw material for basic refractories.

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